

## X. APPENDIX II

### ANALYTICAL METHOD FOR CYCLOHEXANE EXTRACTABLES OF CARBON BLACK

#### Principle of the Method

The cyclohexane-soluble material in the particulates on the glass-fiber and silver membrane filters is extracted with cyclohexane aided by ultrasonication. Blank filters are extracted along with, and in the same manner as, the samples. After extraction, the cyclohexane solution is filtered through a fritted glass funnel. The total material extracted is determined by weighing a dried aliquot of the extract.

#### Range and Sensitivity

When the electrobalance is set at 1 mg, this method can detect 75-2,000  $\mu\text{g}/\text{sample}$ .

#### Precision and Accuracy

When nine aliquots of a benzene solution from a sample of aluminum-reduction plant emissions containing 1,350  $\mu\text{g}/\text{sample}$  were analyzed, the standard deviation was 25  $\mu\text{g}$  [86]. Experimental verification of this method using cyclohexane is not yet complete.

#### Advantages and Disadvantages of the Method

##### (a) Advantages

This procedure is much faster and easier to run than the Soxhlet method.

##### (b) Disadvantages

If the whole sample is not used for cyclohexane-extraction analysis, a small weighing error makes a large error in the final results.

#### Apparatus

- (a) Ultrasonic bath, 90 Kc, 60 watts, partially filled with water.
- (b) Ultrasonic generator, Series 200, 90 Kc, 60 watts.
- (c) Electrobalance capable of weighing to 1  $\mu\text{g}$ .
- (d) Stoppered glass test tube, 150- x 16-mm.

- (e) Teflon weighing cups, 2-ml, approximate tare weight 60 mg.
- (f) Dispensing bottle, 5-ml.
- (g) Pipets, with 0.5-ml graduations.
- (h) Vacuum oven.
- (i) Tweezers.
- (j) Beaker, 50-ml.
- (k) Glassine paper, 3.5- x 4.5-inches.
- (l) Wood application sticks for manipulating filters.
- (m) Funnels, glass-fritted, 15-ml.
- (n) Graduated evaporative concentrator, 10-ml.

#### Reagents

- (a) Cyclohexane, ACS nanograde reagent.
- (b) Chromic acid cleaning solution.
- (c) Acetone, ACS reagent grade.

#### Procedure

(a) All extraction glassware is cleaned with chromic acid cleaning solution, rinsed first with tap water, then with deionized water followed by acetone, and allowed to dry completely. The glassware is rinsed with nanograde cyclohexane before use. The Teflon cups are cleaned with cyclohexane, then with acetone.

(b) Prewrite the Teflon cups to one hundredth of a milligram (0.01 mg).

(c) Remove top of cassette and hold over glassine paper. Remove plug on bottom of cassette. Insert end of application stick through hole and gently raise filters to one side. Use tweezers to remove filters, and loosely roll filters around tweezers. Slide rolled filters into test tube and push them to bottom of tube with application stick. Add any particulates remaining in cassette and on glassine paper to test tube.

(d) Pipet 5 ml of cyclohexane into test tube from dispensing bottle.

- (e) Put test tube into sonic bath so that water level in bath is above liquid level in test tube. A 50-ml beaker filled with water to level of cyclohexane in tube works well.
- (f) Sonify sample for 5 minutes. Do not hold tube in hand while sonifying.
- (g) Filter the extract in 15-ml medium glass-fritted funnels.
- (h) Rinse test tube and filters with two 1.5-ml aliquots of cyclohexane and filter through the fritted-glass funnel.
- (i) Collect the extract and two rinses in the 10-ml graduated evaporative concentrator.
- (j) Evaporate down to 1 ml while rinsing the sides with cyclohexane.
- (k) Pipet 0.5 ml of the extract to preweighed Teflon weighing cup. These cups can be reused after washing with acetone.
- (l) Evaporate to dryness in a vacuum oven at 40 C for 3 hours.
- (m) Weigh the Teflon cup. Use counterweighing technique on electrobalance with full scale range of 1 mg to determine weight of aliquot to nearest microgram. The weight gain is due to the cyclohexane-soluble residue.

#### Calculations

The amount of cyclohexane-extractable fraction present in the sample (in mg) may be determined according to the following equation:

$$\text{mg/sample} = 2 \times [\text{wt sample aliquot (mg)} - \text{wt blank aliquot (mg)}]$$

The amount of cyclohexane-extractable fraction present in the air may then be determined according to the following equation:

$$\text{mg/cu m} = \text{mg/sample} \div \text{air volume collected (cu m)}$$

## XI. APPENDIX III

### MATERIAL SAFETY DATA SHEET

The following items of information which are applicable to a specific product or material shall be provided in the appropriate block of the Material Safety Data Sheet (MSDS).

The product designation is inserted in the block in the upper left corner of the first page to facilitate filing and retrieval. Print in upper case letters as large as possible. It should be printed to read upright with the sheet turned sideways. The product designation is that name or code designation which appears on the label, or by which the product is sold or known by employees. The relative numerical hazard ratings and key statements are those determined by the rules in Chapter V, Part B, of the NIOSH publication, An Identification System for Occupationally Hazardous Materials. The company identification may be printed in the upper right corner if desired.

#### (a) Section I. Production Identification

The manufacturer's name, address, and regular and emergency telephone numbers (including area code) are inserted in the appropriate blocks of Section I. The company listed should be a source of detailed backup information on the hazards of the material(s) covered by the MSDS. The listing of suppliers or wholesale distributors is discouraged. The trade name should be the product designation or common name associated with the material. The synonyms are those commonly used for the product, especially formal chemical nomenclature. Every known chemical designation or competitor's trade name need not be listed.

#### (b) Section II. Hazardous Ingredients

The "materials" listed in Section II shall be those substances which are part of the hazardous product covered by the MSDS and individually meet any of the criteria defining a hazardous material. Thus, one component of a multicomponent product might be listed because of its toxicity, another component because of its flammability, while a third component could be included both for its toxicity and its reactivity. Note that a MSDS for a single component product must have the name of the material repeated in this section to avoid giving the impression that there are no hazardous ingredients.

Chemical substances should be listed according to their complete name derived from a recognized system of nomenclature. Where possible, avoid using common names and general class names such as "aromatic amine," "safety solvent," or "aliphatic hydrocarbon" when the specific name is known.

The "%" may be the approximate percentage by weight or volume (indicate basis) which each hazardous ingredient of the mixture bears to the whole mixture. This may be indicated as a range or maximum amount, ie, "10-40% vol" or "10% max wt" to avoid disclosure of trade secrets.

Toxic hazard data shall be stated in terms of concentration, mode of exposure or test, and animal used, eg, "100 ppm LC50-rat," "25 mg/kg LD50-skin-rabbit," "75 ppm LC man," or "permissible exposure from 29 CFR 1910.1000," or, if not available, from other sources of publications such as the American Conference of Governmental Industrial Hygienists or the American National Standards Institute Inc. Flashpoint, shock sensitivity, or similar descriptive data may be used to indicate flammability, reactivity, or similar hazardous properties of the material.

(c) Section III. Physical Data

The data in Section III should be for the total mixture and should include the boiling point and melting point in degrees Fahrenheit (Celsius in parentheses); vapor pressure, in conventional millimeters of mercury (mmHg); vapor density of gas or vapor (air = 1); solubility in water, in parts/hundred parts of water by weight; specific gravity (water = 1); percent volatiles (indicated if by weight or volume) at 70 F (21.1 C); evaporation rate for liquids sublimable solids, relative to butyl acetate; and appearance and odor. These data are useful for the control of toxic substances. Boiling point, vapor density, percent volatiles, vapor pressure, and evaporation are useful for designing proper ventilation equipment. This information is also useful for design and deployment of adequate fire and spill containment equipment. The appearance and odor may facilitate identification of substances stored in improperly marked containers, or when spilled.

(d) Section IV. Fire and Explosion Data

Section IV should contain complete fire and explosion data for the product, including flashpoint and autoignition temperature in degrees Fahrenheit (Celsius in parentheses); flammable limits, in percent by volume in air; suitable extinguishing media or materials; special firefighting procedures; and unusual fire and explosion hazard information. If the product presents no fire hazard, insert "NO FIRE HAZARD" on the line labeled "Extinguishing Media."

(e) Section V. Health Hazard Information

The "Health Hazard Data" should be a combined estimate of the hazard of the total product. This can be expressed as a TWA concentration, as a permissible exposure, or by some other indication of an acceptable standard. Other data are acceptable, such as lowest LD50 if multiple components are involved.

Under "Routes of Exposure," comments in each category should reflect the potential hazard from absorption by the route in question. Comments should indicate the severity of the effect and the basis for the statement if possible. The basis might be animal studies, analogy with similar products, or human experiences. Comments such as "yes" or "possible" are not helpful. Typical comments might be:

Skin Contact--single short contact, no adverse effects likely; prolonged or repeated contact, possibly mild irritation.

Eye Contact--some pain and mild transient irritation; no corneal scarring.

“Emergency and First Aid Procedures” should be written in lay language and should primarily represent first-aid treatment that could be provided by paramedical personnel or individuals trained in first aid.

Information in the “Notes to Physician” section should include any special medical information which would be of assistance to an attending physician including required or recommended preplacement and periodic medical examinations, diagnostic procedures, and medical management of overexposed employees.

(f) Section VI. Reactivity Data

The comments in Section VI relate to safe storage and handling of hazardous, unstable substances. It is particularly important to highlight instability or incompatibility to common substances or circumstances, such as water, direct sunlight, steel or copper piping, acids, alkalis, etc. “Hazardous Decomposition Products” shall include those products released under fire conditions. It must also include dangerous products produced by aging, such as peroxides in the case of some ethers. Where applicable, shelf life should also be indicated.

(g) Section VII. Spill or Leak Procedures

Detailed procedures for cleanup and disposal should be listed with emphasis on precautions to be taken to protect employees assigned to cleanup detail. Specific neutralizing chemicals or procedures should be described in detail. Disposal methods should be explicit including proper labeling of containers holding residues and ultimate disposal methods such as “sanitary landfill” or “incineration.” Warnings such as “comply with local, state, and Federal antipollution ordinances” are proper but not sufficient. Specific procedures shall be identified.

(h) Section VIII. Special Protection Information

Section VIII requires specific information. Statements such as “Yes,” “No,” or “If necessary” are not informative. Ventilation requirements should be specific as to type and preferred methods. Respirators shall be specified as to type and NIOSH or MSHA approval class, ie, “Supplied air,” “Organic vapor canister,” etc. Protective equipment must be specified as to type and materials of construction.

(i) Section IX. Special Precautions

“Precautionary Statements” shall consist of the label statements selected for use on the container or placard. Additional information on any aspect of safety or health not covered in other sections should be inserted in Section IX. The lower block can contain references to published guides or in-house procedures for handling and storage. Department of Transportation markings and classifications and other freight, handling, or storage requirements and environmental controls can be noted.

(j) Signature and Filing

Finally, the name and address of the responsible person who completed the MSDS and the date of completion are entered. This will facilitate correction of errors and identify a source of additional information.

The MSDS shall be filed in a location readily accessible to employees exposed to the hazardous substance. The MSDS can be used as a training aid and basis for discussion during safety meetings and training of new employees. It should assist management by directing attention to the need for specific control engineering, work practices, and protective measures to ensure safe handling and use of the material. It will aid the safety and health staff in planning a safe and healthful work environment and in suggesting appropriate emergency procedures and sources of help in the event of harmful exposure of employees.

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## MATERIAL SAFETY DATA SHEET

I PRODUCT IDENTIFICATION		
MANUFACTURER'S NAME	REGULAR TELEPHONE NO EMERGENCY TELEPHONE NO	
ADDRESS		
<b>TRADE NAME</b>		
<b>SYNONYMS</b>		
II HAZARDOUS INGREDIENTS		
MATERIAL OR COMPONENT	%	HAZARD DATA
III PHYSICAL DATA		
BOILING POINT, 760 MM HG		MELTING POINT
SPECIFIC GRAVITY (H <sub>2</sub> O = 1)		VAPOR PRESSURE
VAPOR DENSITY (AIR = 1)		SOLUBILITY IN H <sub>2</sub> O, % BY WT
% VOLATILES BY VOL		EVAPORATION RATE (BUTYL ACETATE = 1)
APPEARANCE AND ODOR		

<b>IV FIRE AND EXPLOSION DATA</b>			
FLASH POINT (TEST METHOD)		AUTOIGNITION TEMPERATURE	
FLAMMABLE LIMITS IN AIR, % BY VOL.	LOWER		UPPER
EXTINGUISHING MEDIA			
SPECIAL FIRE FIGHTING PROCEDURES			
UNUSUAL FIRE AND EXPLOSION HAZARD			
<b>V HEALTH HAZARD INFORMATION</b>			
HEALTH HAZARD DATA			
ROUTES OF EXPOSURE			
INHALATION			
SKIN CONTACT			
SKIN ABSORPTION			
EYE CONTACT			
INGESTION			
EFFECTS OF OVEREXPOSURE			
ACUTE OVEREXPOSURE			
CHRONIC OVEREXPOSURE			
EMERGENCY AND FIRST AID PROCEDURES			
EYES			
SKIN			
INHALATION			
INGESTION			
NOTES TO PHYSICIAN			

<b>VI REACTIVITY DATA</b>
CONDITIONS CONTRIBUTING TO INSTABILITY
INCOMPATIBILITY
HAZARDOUS DECOMPOSITION PRODUCTS
CONDITIONS CONTRIBUTING TO HAZARDOUS POLYMERIZATION
<b>VII SPILL OR LEAK PROCEDURES</b>
STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED
NEUTRALIZING CHEMICALS
WASTE DISPOSAL METHOD
<b>VIII SPECIAL PROTECTION INFORMATION</b>
VENTILATION REQUIREMENTS
SPECIFIC PERSONAL PROTECTIVE EQUIPMENT
RESPIRATORY (SPECIFY IN DETAIL)
EYE
GLOVES
OTHER CLOTHING AND EQUIPMENT

**IX SPECIAL PRECAUTIONS**

**PRECAUTIONARY  
STATEMENTS**

**OTHER HANDLING AND  
STORAGE REQUIREMENTS**

**PREPARED BY** \_\_\_\_\_

**ADDRESS** \_\_\_\_\_

**DATE** \_\_\_\_\_

XII. TABLES AND FIGURE

TABLE XII-1

PHYSICAL AND CHEMICAL PROPERTIES OF VARIOUS TYPES OF CARBON BLACK

Property	Furnace Black		Channel Black	Thermal Black
	Oil	Gas		
Composition				
Carbon (%)	98	99.2	88.4-95.2 (avg, 91.2)	—
Oxygen (%)	0.8	0.4	3.6-11.2 (avg, 7.8)	—
Hydrogen (%)	0.3	0.3	0.4-0.8 (avg, 0.6)	—
Ash-Ca, Mg, Na (%)	0.1-1.0	0.1-1.0	0.01	—
Volatile matter (%)	1-2	1-2	5-18	<1
Average particle diameter ( $\mu\text{m}$ )	0.018-0.06	0.04-0.08	0.01-0.03	0.14-0.47
Specific surface (sq m/g)	25-200	25-50	100-1,000	7-13
pH	8-9	8-9	3-5	8-9
Benzene extractables (%)	0.05-0.1	0.05-0.15	—	0.03-1.75

Adapted from reference 2

TABLE XII-2  
OCCUPATIONS WITH POTENTIAL EXPOSURE TO CARBON BLACK

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Battery workers  
Carbon black makers  
Carbon electrode workers  
Carburization workers  
Cement workers  
Ceramics workers  
Food processors  
Ink workers  
Paint manufacture workers  
Paper workers  
Plastics workers  
Printers  
Rubber workers

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Adapted from reference 2

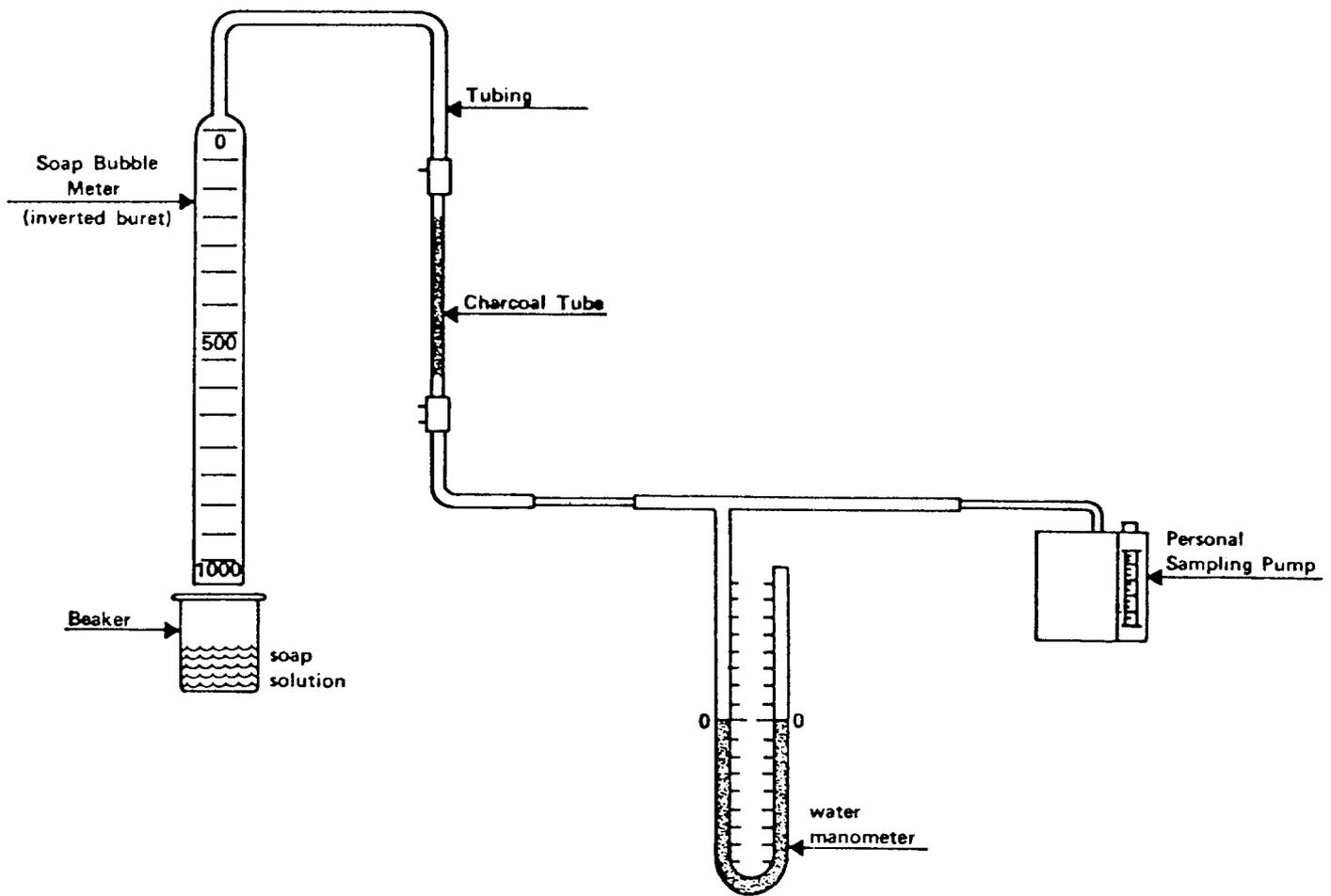


FIGURE XII-1

CALIBRATION SETUP FOR PERSONAL SAMPLING PUMP WITH CHARCOAL TUBE

DEPARTMENT OF  
HEALTH, EDUCATION, AND WELFARE  
PUBLIC HEALTH SERVICE  
CENTER FOR DISEASE CONTROL  
NATIONAL INSTITUTE FOR OCCUPATIONAL SAFETY AND HEALTH  
ROBERT A. TAFT LABORATORIES  
4676 COLUMBIA PARKWAY, CINCINNATI, OHIO 45226

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